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PCT

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<p>(54) Title: POLYVINYL ACETATE ENCAPSULATION OF SUCRALOSE FROM SOLUTIONS FOR USE IN CHEWING GUM</p> <p>(57) Abstract</p> <p>A sucrose-containing chewing gum ingredient is prepared by mixing about 50 to about 95 parts by weight polyvinyl acetate with about 5 to about 50 parts by weight of a solution comprising sucralose dissolved in a solvent. The mixture is then dried to drive off the solvent. A chewing gum is prepared by mixing the sucralose-containing ingredient, gum base, a bulking agent and flavor to make a chewing gum composition.</p>			

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- 1 -

POLYVINYL ACETATE ENCAPSULATION OF  
SUCRALOSE FROM SOLUTIONS FOR USE IN CHEWING GUM

CROSS REFERENCE TO RELATED APPLICATION

This application is a continuation-in-part of Application PCT/US89/05296, filed November 22, 1989 and titled Method of Controlling Release of Sucralose in Chewing Gum and Gum Produced Thereby, the disclosure of which is hereby incorporated by reference.

FIELD OF THE INVENTION

The present invention relates to chewing gum, and more particularly to the use in chewing gum of a polyvinyl acetate encapsulated sucralose from a solution.

BACKGROUND OF THE INVENTION

Chewing gums comprise many ingredients, including sweeteners. Sweeteners suitable for chewing gums include both natural and artificial sweeteners. Recently, high-intensity sweeteners have been the focus of investigation for use in chewing gum.

High-intensity sweeteners may be hundreds of times sweeter than natural sweeteners such as sucrose and glucose. High-intensity sweeteners of recent interest include aspartame, acesulfame K, cyclamates, saccharin, and now sucralose. Sucralose is a new, high-intensity sweetener which is a tri-chlorinated sucrose derivative and is about 400-800 times sweeter than sucrose. Sucralose has been reported to be cheaper than aspartame and at least 3 times sweeter. Compared to some other high-intensity sweeteners, sucralose has a sweet taste more similar to sucrose. Chemically, sucralose is known as 4,1',6'-trichloro-4,1',6'-trideoxy-galactosucrose, or

- 2 -

alternatively 1,6'-dichloro-1,6-dideoxy-( $\beta$ )-D-fructofuranosyl 4-chloro-4-deoxy-( $\alpha$ )-D-galactopyranoside. The United States Food and Drug Administration is now considering sucralose as a food additive. The manufacturer of sucralose is Tate & Lyle, Inc. in the U.K. Sucralose is marketed in the United States by McNeil Specialty Products Co., Skillman, New Jersey. Its use to sweeten substances, including oral compositions, is disclosed in U.S. Patent Nos. 4,343,934 and 4,389,394, both of which are incorporated herein by reference. The use of sucralose as an artificial sweetener is also disclosed in U.K. Patent Nos. 2,065,646; 2,065,648 and 2,153,651.

The use of sucralose in chewing gum has been disclosed in other patents.

U.S. Patent No. 4,495,170 discloses sweetener mixtures for use in foods, beverages and pharmaceuticals but not specifically chewing gums. The mixtures are of sucralose and aspartame, saccharin, and acesulfame K. U.S. Patent No. 4,389,394 discloses the use of artificial sweeteners in chewing gum to prevent dental caries. Chlorinated sucrose derivatives are disclosed in U.S. Patent Nos. 4,435,440; 4,549,013; and 4,612,373.

U.K. Patent No. 2,154,850 discloses beverages sweetened with sucralose and cyclamate (among others).

U.K. Patent No. 2,185,674 discloses combinations of thaumatin and sucralose (among others) to stimulate growth of farm animals.

U.S. Patent No. 4,820,528 discloses co-dried sucralose and acid saccharin to prolong sweetness release.

One of the limitations of chewing gum is that the sweetness and flavor are rapidly lost during chewing. Thus, chewing gum producers are constantly trying to extend the time in which a consumer of chewing gum can enjoy its flavor and sweetness.

Patents disclose how a sweetener like aspartame can be physically modified to control the release rate in chewing gum.

- 3 -

For example, U.S. Patent No. 4,597,970 to Sharma et al. teaches a process for producing an agglomerated sweetener wherein the sweetener is dispersed in a hydrophobic matrix consisting essentially of lecithin, a glyceride, and a fatty acid or wax having a melting point between 25 and 100°C. The method disclosed uses a spray-congealing step to form the sweetener-containing matrix into droplets, followed by a fluid-bed second coating on the agglomerated particles.

U.S. Patent Nos. 4,515,769 and 4,386,106, both to Merrit et al., teach a two step process for preparing a delayed release flavorant for chewing gum. In this process, the flavorant is prepared in an emulsion with a hydrophilic matrix. The emulsion is dried and ground and the particles are then coated with a water-impermeable substance.

U.S. Patent No. 4,230,687 to Sair et al. teaches a process for encasing an active ingredient to achieve gradual release of the ingredient in a product such as chewing gum. The method described involves adding the ingredient to an encapsulating material in the form of a viscous paste. High-shear mixing is used to achieve a homogeneous dispersion of the ingredient within the matrix, which is subsequently dried and ground.

U.S. Patent No. 4,139,639 to Bahoshy et al. teaches a process of "fixing" aspartame by co-drying (by spray drying or fluid-bed coating) a solution containing aspartame and an encapsulating agent, such as gum arabic, to thereby surround and protect the aspartame in the gum during storage.

U.S. Patent No. 4,384,004 to Cea et al. teaches a method of encapsulating aspartame with various solutions of encapsulating agents using various encapsulation techniques, such as spray drying, in order to increase the shelf-stability of the aspartame.

U.S. Patent No. 4,634,593 to Stroz et al. teaches a method for producing controlled release sweeteners for confections, such as chewing gum. The method taught therein involves the use of an insoluble fat material which is mixed with the sweetener.

- 4 -

PCT Publication WO 90/07859 discloses a number of techniques, including encapsulation techniques, for delaying the release of sucralose from chewing gum. In example 18 of the publication, sucralose is mixed with polyvinyl acetate and the combined material is added to other gum base ingredients and made into gum.

It is known that the sweetness and flavor can be extended in chewing gum by using high levels of high intensity sweeteners such as aspartame. The extension properties of those sweeteners, however, are limited by their release characteristics during chewing of the gum. Providing a chewing gum having improved sweetness and flavor extension is one object of this invention.

Sucralose, when modified according to the present invention and incorporated into chewing gum, gives a chewing gum having a controlled-release sweetener. A higher quantity of sweetener can be used without resulting in a high initial sweetness impact, but instead having a delayed sweetness release, giving a highly consumer-acceptable chewing gum product.

#### SUMMARY OF THE INVENTION

The present invention includes sucralose-containing chewing gum ingredients and methods of making the ingredients. The methods include the steps of homogeneously mixing 50 to 95 parts by weight polyvinyl acetate (PVAc) with 5 to 50 parts by weight of a solution prepared by dissolving sucralose in a solvent. The homogeneous mixture is then dried to drive off the solvent, leaving a blended, dried ingredient.

The invention also includes chewing gum compositions containing the sucralose-containing ingredients and methods of making the gum. The gum making methods include the steps of providing the sucralose-containing ingredient as outlined above, and mixing the same with gum base, a bulking agent and flavoring agent to make a chewing gum composition.

It has been found that the sucralose incorporated in chewing gum using the sucralose-containing ingredient of the

- 5 -

present invention is surprisingly slow to release from the gum. Thus the invention provides a very simple but effective delay release mechanism for sucralose.

DETAILED DESCRIPTION OF THE  
PREFERRED EMBODIMENTS OF THE INVENTION

As used herein, the term "chewing gum" includes chewing gum, bubble gum and the like. Unless specified otherwise, all percents used herein are weight percents.

The first step in preparing the sucralose-containing ingredient of the present invention is to dissolve the sucralose in a solvent. The solubility of sucralose in water, the preferred solvent, is approximately 26% at 20°C, and increases with temperature. Sucralose is soluble in ethanol up to 30%, and 20% is readily soluble. Sucralose is also soluble in methanol. These and other solvents may be used as long as the level of solvent remaining when the ingredient is added to the gum is acceptable for a food ingredient, the solvent is compatible with PVAc, and the solvent will dissolve sucralose at an acceptable level. The solvent may also be a solvent for PVAc and this may be a benefit for some mixing methods. The level of sucralose in the solvent will depend on the solubility of sucralose in the solvent, the level of solvent compatible with PVAc, and the desired level of sucralose in the finished ingredient. Where the solvent is water, the concentration may range from 5 to 60%. Levels above 25% will require mild heating to keep the sucralose in solution.

The next step is to homogeneously mix PVAc and the sucralose solution. The PVAc used in the present invention will be a food grade polymer. The molecular weight of the polymer is not believed to be critical to working of the invention, but certain molecular weights may be easier to use to produce the blended ingredient and others may be more effective at extending the release rate of sucralose. Specifically, lower molecular weights of PVAc tend to be easier to mix and reduce the effort necessary to clean out the

- 6 -

mixing apparatus afterwards. However, there is some evidence that higher molecular weights provide slightly slower release.

The contemplated molecular weight range of PVAc usable in the present invention is from about 7000 to about 100,000 molecular weight units (MWU). The preferred range is from about 10,000 MWU to about 50,000 MWU and most preferred are polymers of about 15,000 MWU to about 32,000 MWU. (The preceding molecular weight numbers are based on average molecular weight as measured by Gel Permeation Chromatography. Other methods of measuring molecular weights of polymers may give substantially different values.)

The level of sucralose used in the PVAc blend can vary greatly. An upper limit would be just below the amount where the sucralose loading is so high that the PVAc can no longer form a film upon drying. Levels in the range of about 3% to about 50% are specifically contemplated, while about 15% to about 35% is preferred. The most preferred level is about 20% to about 30%. The exact level chosen will depend on the intended usage level of this ingredient in the final gum composition.

A variety of methods of preparing the sucralose blends of the present invention are contemplated. The PVAc/sucralose blend may be mixed in a conventional sigma blade gum mixer. Any other type of mixer suitable for the task may also be used. Usually heat will be used to soften the PVAc to aid mixing. Preferred temperatures for mixing are in the range of 100 to 212°F. The PVAc may also be softened by the solvent used in the solution. For example, PVAc is soluble in ethanol up to 55% at 101°F and 40% at 70°F. Thus, mixing sucralose with ethanol and adding the solution to ground PVAc will yield a homogeneous blend of PVAc, ethanol and sucralose.

Regardless of the solvent used, the solution can only be added up to the point where no more solution will homogeneously mix with the softened or melted PVAc. This level will depend primarily on the solvent. When water is used as the solvent, it was found that only about 25 grams of

- 7 -

a 25% sucralose solution could be added per 100 grams of PVAc before the solution would no longer mix in, but remain as a separate phase on top of the PVAc.

One specifically contemplated mixing apparatus is a vacuum kettle with one or more blades. With relatively high heating and vacuum, a continuous solution addition and continuous evaporation to achieve higher loading should be possible. However, final drying is best carried out in a separate apparatus.

After the mixing process, the blended ingredient must be dried to remove the solvent. This may be accomplished by drying in a vacuum over at 100° to 212°F (in the case of an aqueous solution). It is desirable to keep final drying temperatures below 150°F to prevent sucralose degradation. The preferred temperature is 130°F. Forced air ovens or other suitable drying apparatus or methods may be used. The final solvent level may range from about 0.1% to about 10%. When water is used as the solvent, a final moisture level of about 0.5% to about 2% is preferred. It is believed that the final moisture level will directly affect the ability of the ingredient to provide a delayed release. Currently it is believed that lower moisture levels will result in more delayed release of the sucralose. However, it is possible that higher moisture levels may operate effectively if the ingredient moisture migrates into the gum during storage. This would allow the use of higher initial ingredient moisture levels.

After drying, the ingredient is preferably sized, usually by grinding and/or sieving to provide the desired particle size. The particle size should be small enough so as not to be noticeable in the finished gum. Excessively fine particles may lose some of the slow release benefit, but in practice, this is not a serious problem. The preferred particle size is less than about 420 microns. Usually a size range of about 80 microns to about 400 microns is desirable.

The usage level of the encapsulated ingredient in gum will vary with the active level of sucralose in the

- 8 -

encapsulation. In general, the usage level will be calculated to give a desired level of active sucralose in the gum. Usage levels of the encapsulated ingredient of about 0.02 percent to about 20 percent are therefore contemplated. The actual optimized usage level may be higher than might be theoretically calculated due to the slower release rate of the blended ingredient. Depending on the desired sweetness level and the presence of other sweeteners in the gum, the active sucralose level may vary from about 0.01% to about 0.50% or even higher.

In general, a chewing gum composition typically comprises a water-soluble bulk portion, a water-insoluble chewable gum base portion and typically water-insoluble flavoring agents. The water-soluble portion dissipates with a portion of the flavoring agent over a period of time during chewing. The gum base portion is retained in the mouth throughout the chew.

The insoluble gum base generally comprises elastomers, resins, fats and oils, waxes, softeners and inorganic fillers. Elastomers may include polyisobutylene, isobutylene-isoprene copolymer and styrene butadiene rubber, as well as natural latexes such as chicle. Resins include polyvinyl acetate and terpene resins. Fats and oils may also be included in the gum base, including tallow, hydrogenated and partially hydrogenated vegetable oils, and cocoa butter. Commonly employed waxes include paraffin, microcrystalline and natural waxes such as beeswax and carnauba. According to the preferred embodiment of the present invention, the insoluble gum base constitutes between about 5 to about 80 percent by weight of the gum. More preferably the insoluble gum base comprises between 10 and 50 percent by weight of the gum and most preferably about 20 to about 35 percent by weight of the gum.

The gum base typically also includes a filler component. The filler component may be calcium carbonate, magnesium carbonate, talc, dicalcium phosphate or the like. The filler may constitute between about 5 and about 60 percent

- 9 -

by weight of the gum base. Preferably, the filler comprises about 5 to about 50 percent by weight of the gum base.

Gum bases typically also contain softeners, including glycerol monostearate and glycerol triacetate. Further, gum bases may also contain optional ingredients such as antioxidants, colors, and emulsifiers. The present invention contemplates employing any commercially acceptable gum base.

The water-soluble portion of the chewing gum may further comprise softeners, sweeteners, flavoring agents and combinations thereof. Softeners are added to the chewing gum in order to optimize the chewability and mouth feel of the gum. Softeners, also known in the art as plasticizers or plasticizing agents, generally constitute between about 0.5 to about 15.0 percent by weight of the chewing gum. Softeners contemplated by the present invention include glycerin, lecithin, and combinations thereof. Further, aqueous sweetener solutions such as those containing sorbitol, hydrogenated starch hydrolysates, corn syrup and combinations thereof may be used as softeners and binding agents in gum.

The sucralose-containing ingredient of the present invention will most likely be used in sugarless gum formulations. However, formulations containing sugar are also within the scope of the invention. Sugar sweeteners generally include saccharide-containing components commonly known in the chewing gum art which comprise, but are not limited to, sucrose, dextrose, maltose, dextrin, dried invert sugar, fructose, levulose, galactose, corn syrup solids and the like, alone or in any combination. Generally sugarless sweeteners include components with sweetening characteristics but which are devoid of the commonly known sugars and comprise, but are not limited to, sugar alcohols such as sorbitol, mannitol, xylitol, hydrogenated starch hydrolysates, maltitol and the like, alone or in any combination.

These sugar and sugarless sweeteners often also serve the role as bulking agents in chewing gum. The bulking

- 10 -

agent generally comprises from about 5 percent to about 80 percent of the gum composition.

Depending on the particular sweetness release profile and shelf-stability needed, the sucralose-containing ingredient of the present invention can also be used in combination with uncoated high-potency sweeteners or with high-potency sweeteners coated with other materials and by other techniques. In particular, sucralose treated to increase its release rate as described in U.S. Patent Application Serial No. 07/376,037, incorporated herein by reference, may be used in conjunction with the present invention.

A flavoring agent may be present in the chewing gum in an amount within the range of from about 0.1 to about 10.0 weight percent and preferably from about 0.25 to about 5 weight percent of the gum. The flavoring agents may comprise essential oils, synthetic flavors, or mixture thereof including, but not limited to, oils derived from plants and fruits such as citrus oils, fruit essences, peppermint oil, spearmint oil, clove oil, oil of wintergreen, anise, and the like. Artificial flavoring components are also contemplated for use in gums of the present invention. Those skilled in the art will recognize that natural and artificial flavoring agents may be combined in any sensorially acceptable blend. All such flavors and flavor blends are contemplated by the present invention.

Optional ingredients such as colors, emulsifiers and pharmaceutical agents may be added to the chewing gum.

In general, chewing gum is manufactured by sequentially adding the various chewing gum ingredients to a commercially available mixer known in the art. After the ingredients have been thoroughly mixed, the gum mass is discharged from the mixer and shaped into the desired form, such as by rolling into sheets and cutting into sticks, extruding into chunks or casting into pellets.

Generally, the ingredients are mixed by first melting the gum base and adding it to the running mixer. The

- 11 -

base may also be melted in the mixer itself. Color or emulsifiers may also be added at this time. A softener such as glycerin may also be added at this time, along with syrup and a portion of the bulking agent. Further portions of the bulking agent may then be added to the mixer. A flavoring agent is typically added with the final portion of the bulking agent.

The entire mixing procedure typically takes from five to fifteen minutes, but longer mixing times may sometimes be required. Those skilled in the art will recognize that many variations of the above described procedure may be followed.

The blended sucralose-containing ingredient may be added at any point in the gum making process. The preferred point is with the first bulk sweetener addition. Very early addition into hot base is undesirable since the PVAc may soften or melt.

Example 1:

A sucralose-containing ingredient was prepared by mixing 100g of ground and softened 15,000 MW PVAc with 30g of 25% aqueous sucralose solution. The mixing was carried out in a small capacity lab mixer heated to 112°F. The softened PVAc absorbed approximately 22g of the solution, the remainder being lost through the shaft seals. The mixture was dried in a convection oven at 130°F for 16 hours. The final moisture was 0.6% and the loading, by analysis, was 4.8%. The ingredient was ground in a Fitzmill grinder with a 0.109 inch screen. The ground ingredient was sieved and the fraction between 40 and 325 U.S. Standard mesh size was used.

Example 2:

The method of Example 1 was repeated except that after drying and grinding, the product was softened by heating and subjected to mixing with additional quantities of solution, then dried again. The PVAc/sucralose blend was then ground and sieved as before. The final moisture was 1.4% and the analytical sucralose content was 12.3%.

- 12 -

Example 3 (Comparative Example):

The method of Example 1 was repeated except that a 25% aqueous acesulfame K solution was substituted for the sucralose solution. The final moisture was 1.58% and the level of acesulfame K in the blended, dried ingredient was calculated to be 6.2%.

Examples 4, 5, 6, 7 (Chewing Gums):

Chewing gums were made according to the following formulas (in percent by weight).

	<u>Example 4</u>	<u>Example 5</u>	<u>Example 6</u>	<u>Comparative Example 7</u>
Sugar	52.720	53.164	52.790	53.695
Base	22.255	22.255	22.255	22.255
Corn Syrup	5.130	5.130	5.130	5.130
Glycerin	2.000	2.000	2.000	2.000
Calcium Carbonate	2.000	2.000	2.000	2.000
10% Salt Solution	0.100	0.100	0.100	0.100
Dextrose	13.410	13.410	13.410	13.410
Spearmint Flavor	1.350	1.350	1.350	1.350
Ingredient of Ex. 1	1.035	-	-	-
Ingredient of Ex. 2	-	0.591	-	-
Ingredient of Ex. 3	-	-	0.965	-
Crystalline Sucralose	-	-	-	0.060
	100.000	100.00	100.000	100.000
Active Sweetener	0.0497	0.0727	0.0598*	0.0600

\* Based on theoretical level of acesulfame K in the PVAc blend (other levels based on analytical values).

Note that Example 7 is a practicing of one aspect of the invention disclosed in U.S. Patent Application Serial No. 07/376,037 as the base composition is one which was found to delay the release of sucralose.

Sensory Testing

The gums of Examples 5 and 7 were compared in a 20 minute chew for sweetness duration. Six trained panelists chewed the samples along with two other samples not related to the present test. The samples were blind coded and presented in random order. The panelists ranked the samples from greatest to least sweetness duration. The panelists also made additional comments on the samples. The average rankings (1 being the longest-lasting, 4 the shortest) were:

- 13 -

Average  
Order Ranking

Gum of Example 5 (inventive)	2.0
Gum of Example 7 (control)	3.5

Panelists noted a faster decline of sweetness and flavor intensity in Example 7 and fluctuations in the late sweetness level in both samples. These results show that the inventive ingredient performs well when compared to sucralose mixed directly into gum with a base composition known to delay the release of the sucralose.

Analytical Release Rate

The gums of Examples 4, 5 and 6 were chewed by several individuals for 0, 2, 5, 10 and 20 minutes. The cuds were collected and analyzed for residual sweetener to determine the release rates. The results are tabulated below.

- 14 -

TABLE 1

Chew Out Test Results

Example 4 gum (4.8% Loading):

	<u>Level</u> <u>%</u>	<u>% of Orig.</u> <u>Remaining</u>
Theoretical (0 min.)	0.0497	-
Analyzed (0 min.)	0.056	100
2 min.	0.045	80
5 min.	0.045	80
10 min.	0.044	79
20 min.	0.027	48

Example 5 gum (12.3% Loading):

	<u>Level</u> <u>%</u>	<u>% of Orig.</u> <u>Remaining</u>
Theoretical (0 min.)	0.0727	-
Analyzed (0 min.)	0.074	100
2 min.	0.080	108
5 min.	0.062	84
10 min.	0.066	89
20 min.	0.042	57

Example 6 gum (Acesulfame K, 6.2% Loading):

	<u>Level</u> <u>%</u>	<u>% of Orig.</u> <u>Remaining</u>
Theoretical (0 min.)	0.0598	-
Analyzed (0 min.)	0.035	100
2 min.	0.013	37
5 min.	0.009	26
10 min.	0.003	9
20 min.	0.001	3

In previous tests with similar gum formulas, with crystalline sucralose mixed directly into the gum, some of which are disclosed in Application Serial No. 07/376,037, the gum retained approximately 20% of its original sucralose level after 20 minutes of chewing. Ethanol and aqueous solutions of sucralose mixed directly into gum produced gums that retained less than 10% of their original level after 20 minutes of chewing. Thus, in comparison, the results in Table 1 show

- 15 -

that the sucralose encapsulated from solution by PVAc has a significantly delayed release from chewing gum when compared to these other methods of mixing sucralose into gum. In fact, the present invention is more effective in prolonging release than the slow release base formulas previously disclosed.

The results of Table 1 also show that the invention is uniquely suited for sucralose encapsulation. When acesulfame K, a sweetener with similar water solubility, is subjected to the same treatment steps, the resulting gum does not exhibit the same surprising delayed release of the acesulfame K. In fact, the release was found to be no slower than when acesulfame K is mixed directly into gum.

Other methods of forming the sucralose-containing ingredient are given in the following example.

Example 8

Crystalline sucralose dissolved in ethanol at a level of 20% is mixed with PVAc dissolved in ethanol at a level of 40%. The ratio of sucralose solution to PVAc solution is 1:2. The solutions are thoroughly mixed and the ethanol is then evaporated in a safe manner. The resulting product will have a loading of 20%.

Example 9

Crystalline sucralose is dissolved at 20% in ethanol. 100 grams of solution is mixed with 20 grams of ground PVAc have an average molecular weight of 15,000 MWU. The ethanol softens the PVAc, allowing a homogenous mixture to be made. The ethanol is evaporated, leaving 40 grams of a sucralose-containing ingredient having a loading of 50%.

In addition to slowing the release rate of sucralose from gum during chewing, the present invention offers certain other practical benefits. The volume of material added is increased by the bulking effect of the PVAc, allowing easier and more homogeneous mixing into the gum. The sucralose solution is stable, allowing long-term storage of the sucralose in this form prior to manufacture of the PVAc blend.

- 16 -

(An anti-microbial agent or pH buffering agent should be used in aqueous solutions. Such a solution [at 25% sucralose concentration] is available experimentally from McNeil Specialty Product Company in the U.S.) This is in contrast to dry, crystalline sucralose, which is somewhat unstable. The PVAc/sucralose ingredient is also stable, both when stored as an ingredient and in the gum.

It should be appreciated that the compositions and methods of the present invention are capable of being incorporated in the form of a variety of embodiments, only a few of which have been illustrated and described above. The invention may be embodied in other forms without departing from its spirit or essential characteristics. The described embodiments are to be considered in all respects only as illustrative and not restrictive, and the scope of the invention is, therefore, indicated by the appended claims rather than by the foregoing description. All changes which come within the meaning and range of equivalency of the claims are to be embraced within their scope.

- 17 -

We Claim:

1. A method of producing chewing gum comprising the steps of:

a) providing a blended, dried sucralose-containing ingredient by

i) homogeneously mixing 50 to 95 parts by weight PVAc with 5 to 50 parts by weight of a solution comprising sucralose and a solvent and

ii) drying the homogenous mixture to drive off the solvent; and

b) mixing

i) about 0.02% to about 20% by weight of the chewing gum of the blended, dried sucralose-containing ingredient,

ii) about 5% to about 80% by weight of the chewing gum of a base,

iii) about 5% to about 80% by weight of the chewing gum of a bulking agent, and

iv) about 0.25% to about 5% by weight of a flavoring agent to make the chewing gum composition.

2. The method of Claim 1 wherein the PVAc is softened by heating the PVAc before mixing it with the solution.

3. The method of Claim 1 wherein the PVAc is softened by the solvent used in the solution.

4. The method of Claim 1 further comprising the step of sizing the blended, dried ingredient to a particle size small enough to be unnoticeable when mixed into a chewing gum composition.

5. The method of Claim 1 wherein the blended, dried ingredient has a sucralose level between about 3% and about 50%.

- 18 -

6. The method of Claim 1 wherein the blended, dried ingredient has a sucralose level of between about 15% and about 35%.

7. The method of Claim 1 wherein the blended, dried ingredient has a sucralose level of between about 20% to about 30%.

8. The method of Claim 1 wherein the PVAc has an average molecular weight of between about 7000 MWU and about 100,000 MWU as measured by Gel Permeation Chromatography.

9. The method of Claim 1 wherein the PVAc has an average molecular weight of between about 10,000 MWU and about 50,000 MWU as measured by Gel Permeation Chromatography.

10. The method of Claim 1 wherein the solvent is water.

11. The method of Claim 1 wherein the solution comprises about 5% to about 60% sucralose.

12. The method of Claim 1 wherein the solution is heated prior to mixing with the PVAc to maintain the sucralose in a dissolved state.

13. The method of Claim 1 wherein the drying temperature is between about 100°F and 212°F.

14. The method of Claim 1 wherein the drying temperature is below about 150°F.

15. The method of Claim 1 wherein the solvent content of the blended, dried ingredient is below about 10%.

16. The method of Claim 10 wherein the solvent content of the blended, dried ingredient is between about 0.5% and about 2%.

- 19 -

17. The method of Claim 4 wherein the blended, dried ingredient is sized to less than about 420 microns.

18. The method of Claim 4 wherein the blended, dried ingredient is sized to between about 80 microns and about 400 microns.

19. The method of Claim 1 wherein the level of sucralose in the gum is between about 0.01% and about 0.5%.

20. A method of making a sucralose-containing ingredient for chewing gum comprising the steps of:

- a) homogeneously mixing 50 to 95 parts by weight PVAc with 5 to 50 parts by weight of a solution comprising sucralose and a solvent, and
- b) drying the homogenous mixture to drive off the solvent, leaving a blended, dried ingredient.

21. The method of Claim 20 further comprising the step of sizing the blended, dried ingredient to a particle size small enough to be unnoticeable when mixed into a chewing gum composition.

22. A chewing gum composition made in accordance with the method of Claim 1.

# INTERNATIONAL SEARCH REPORT

International Application No PCT/US90/06835

## I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) <sup>3</sup>

According to International Patent Classification (IPC) or to both National Classification and IPC  
**IPC(5): A23G 3/30**  
**US CL : 426/3,4,5,6,96,548,658,804**

## II. FIELDS SEARCHED

Minimum Documentation Searched <sup>4</sup>

Classification System	Classification Symbols
US	426/3,4,5,6,96,548,658,804
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched <sup>5</sup>	

## III. DOCUMENTS CONSIDERED TO BE RELEVANT <sup>14</sup>

Category <sup>6</sup>	Citation of Document, <sup>15</sup> with indication, where appropriate, of the relevant passages <sup>17</sup>	Relevant to Claim No. <sup>18</sup>
A	US, A, 4,612,373 (KAHN ET AL) 16 SEPTEMBER 1986	1-22
A	US, A, 4,343,934 (JENNER ET AL) 10 AUGUST 1982	1-22
A	US, A, 4,384,004 (CEA ET AL) 17 MAY 1983	1-22
A	US, A, 4,405,654 (LEE) 20 SEPTEMBER 1983	1-22
A	US, A, 4,495,170 (BEYTS ET AL) 22 JANUARY 1985	1-22

### \* Special categories of cited documents: <sup>16</sup>

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

## IV. CERTIFICATION

Date of the Actual Completion of the International Search <sup>19</sup>:

08 FEBRUARY 1991

International Searching Authority <sup>20</sup>:

ISA/US

Date of Mailing of the International Search Report <sup>21</sup>:

05 MAR 1991

Signature of Authorized Officer  
**JEANETTE M. HUNTER**  
**INTERNATIONAL DIVISION**  
*Nguyen*

**FURTHER INFORMATION CONTINUED FROM THE SECOND SHEET**

A	US, A, 4,820,528 (STROZ ET AL) 11 APRIL 1989	1-22
A	US, A, 4,435,440 (HOUGH ET AL) 06 MARCH 1984	1-22
A	US, A, 4,549,013 (HOUGH ET AL) 22 OCTOBER 1985	1-22

V.  OBSERVATIONS WHERE CERTAIN CLAIMS WERE FOUND UNSEARCHABLE<sup>1</sup>

This international search report has not been established in respect of certain claims under Article 17(2) (a) for the following reasons:

1.  Claim numbers , because they relate to subject matter not required to be searched by this Authority, namely:

2.  Claim numbers . . . , because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out<sup>1</sup>, specifically:

3.  Claim numbers \_\_\_\_\_, because they are dependent claims not drafted in accordance with the second and third sentences of PCT Rule 6.4(a).

**VI.  OBSERVATIONS WHERE UNITY OF INVENTION IS LACKING<sup>2</sup>**

This International Searching Authority found multiple inventions in this international application.

1.  As all required additional search fees were timely paid by the applicant, this international search report covers all claims of the international application.

2.  As only some of the required additional search fees were timely paid by the applicant, this international search report covers those claims of the international application for which fees were paid, specifically claims:

3.  No required additional search fees were timely paid by the applicant. Consequently, this international search was not conducted for the invention first mentioned in the claims; it is covered by claim numbers:

4.  As all searchable claims could be searched without effort justifying an additional fee, the International Search Act. invite payment of any additional fee.

### **Remark on Protest**

The additional search fees were accompanied by applicant's protest.  
 No protest accompanied the payment of additional search fees.